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METHODS OF TEST FOR NATURAL AND SYNTHETIC PERFUMERY MATERIALS

PART 11 DETERMINATION OF CARBONYL VALUE AND CONTENT OF CARBONYL COMPOUNDS

(Second Revision)

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METHODS OF TEST FOR NATURAL AND SYNTHETIC PERFUMERY MATERIALS

PART 11 DETERMINATION OF CARBONYL VALUE AND CONTENT OF CARBONYL COMPOUNDS

(Second Revision)

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Indian Standard

METHODS OF TEST FOR NATURAL AND SYNTHETIC PERFUMERY MATERIALS

PART 11 DETERMINATION OF CARBONYL VALUE AND CONTENT OF CARBONYL COMPOUNDS

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 15 October 1986, after the draft finalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1952 entitled 'Methods of test for essential oils' and subsequently revised in 1968 as 'Methods of sampling and test for natural and synthetic perfumery materials'. Taking cognizance of the need to incorporate instrumental methods of analysis now in vogue and to facilitate inclusion of additional test methods or changes in the existing test methods and also to align these test methods with the corresponding ISO Standards, the Committee decided to revise and split the standard and publish individual test methods as separate parts of the original standard.

0.2.1 The test method as given in this part supersedes the test method as given under **13** of IS : 326-1968*. An amendment in this respect is being issued separately to the relevant clause of IS : 326-1968*.

0.3 This standard is essentially based on:

a) ISO 1271-1983 Essential oils — Determination of carbonyl value — Free hydroxylamine method; and
b) ISO 1279-1973 Essential oils — Determination of carbonyl compounds content — Hydroxylammonium chloride method, published by International Organization for Standardization.

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*Methods of sampling and test for natural and synthetic perfumery materials (*first revision*).

0.4 In reporting the result of a test or analysis, made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960.*

1. SCOPE

1.1 This standard prescribes two methods, namely, hydroxylammonium chloride and free hydroxylamine methods for the determination of carbonyl value of a perfumery material by the use of oximation.

2. TERMINOLOGY

2.1 For the purpose of this standard the definitions given in IS : 6597-1972† shall apply.

2.2 Carbonyl Value — The carbonyl value of a perfumery material is the mass of potassium hydroxide in mg that is an equivalent to the mass of hydroxylamine required to oximate the carbonyl compounds present in 1 g of the oil.

3. DETERMINATION OF CARBONYL VALUE

3.0 General — Two methods, namely, hydroxylammonium chloride method and free hydroxylamine method are available for determining the carbonyl value of a perfumery material. Any one of these may be adopted for the determination of carbonyl value of a perfumery material, as specified in the individual standard or as may be agreed to between the purchaser and the supplier. The procedure for the two methods have been prescribed under **3.1** and **3.2** respectively.

Note — It may be pointed out that each of the general methods for determination as carbonyl value has its place in the analysis of natural and synthetic perfumery materials. Large difference may be indicative of adulteration with water soluble constituents or additions of carbonyl compounds of low molecular weight. From a consideration of the limitations of each method, it should be obvious that it is of utmost importance always to record the method used when reporting an analytical result.

3.1 Hydroxylammonium Chloride Method — Method A

3.1.0 *Outline of Method* — The carbonyl compounds to be determined are converted into oximes by reaction with hydroxylammonium chloride. The hydrochloric acid liberated during the reaction is determined by an ethanolic potassium hydroxide solution.

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*Rules for rounding off numerical values (*revised*).

†Glossary of terms relating to natural and synthetic perfumery materials.

3.1.1 *Apparatus*

3.1.1.1 *Conical flask* — 200-ml capacity, with ground-glass stopper.

3.1.1.2 *Graduated cylinder* — 25-ml capacity (*see* IS : 878-1975*).

3.1.1.3 *Burette* — of 25-ml (*see* IS : 1997-1967†).

3.1.2 *Reagents*

3.1.2.1 *Potassium hydroxide* — Approximately 0·5 N solution in 95 percent (*v/v*) ethanol, standardized immediately before use against 0·5 N hydrochloric acid, using bromophenol blue as indicator and running the alkali into the acid.

3.1.2.2 *Bromophenol blue indicator solution* — Dissolve, while warming, 0·2 g of bromophenol blue in 3 ml of ethanolic potassium hydroxide, 0·1 N and 10 ml of ethanol 95 percent (*v/v*). After cooling to room temperature make up to 100 ml with ethanol of the same strength.

3.1.2.3 *Hydroxylammonium chloride, ethanolic solution* — Dissolve 50 g of hydroxylammonium chloride in approximately 100 ml of water, and about 800 ml of 95 percent (*v/v*) ethanol, then 10 ml of the ethanolic bromophenol blue solution (**3.1.2.2**) and dilute to 1 000 ml with 95 percent (*v/v*) ethanol. Add the ethanolic potassium hydroxide solution (**3.1.2.1**) until the solution is green, if the liquid is observed in a thin layer, or until red, if the layer is thick.

This solution is suitable for use if a lemon-yellow colour is obtained when 0·05 ml of the hydrochloric acid is added to 20 ml of the solution, and a red colour is obtained when 0·05 ml of the potassium hydroxide solution (**3.1.2.1**) is added to another 20 ml of the solution.

The solution is stable for 1 week.

3.1.3 *Procedure* — Weigh, to the nearest 1 mg, into the conical flask the amount of perfumery material specified in the individual standard. Add from the graduated cylinder 25 ml of hydroxylammonium chloride solution and set aside for the time specified in individual standard. If additional heating is necessary, this will be specified in individual standard.

After the time prescribed, and if necessary after cooling to room temperature, titrate the contents of the flask with the ethanolic potassium hydroxide solution, taking care to avoid going beyond the greenish-yellow colour of the indicator, continue the titration with the potassium hydroxide solution until a bluish green colour persisting for 5 minutes is obtained.

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*Specification for graduated measuring cylinder.

†Specification for burettes (*first revision*).

NOTE — In the case of perfumery material like oil of vetiver where the acid value of the oil is high, prior neutralization of the free acids shall be carried out by taking the required amount of oil (0·3 g is normally taken), accurately weighed and dissolving in 20 ml of neutral alcohol and neutralizing against 0·5 N alcoholic potassium hydroxide solution.

3.1.4 *Calculation* — The carbonyl value and content of carbonyl compounds are calculated as follows:

3.1.4.1 Carbonyl value $= \dfrac{56{\cdot}1 \times V \times N}{m}$

where

V = volume in ml of ethanolic potassium hydroxide solution used in the determination;

N = the normality of the ethanolic potassium hydroxide solution used; and

m = mass in g of the sample.

3.1.4.2 Content of carbonyl compounds (as aldehyde or ketone), percent by mass

$$= \frac{M \times V \times N}{10\,m}$$

where

M = molecular mass of the carbonyl compounds in which the results are to be expressed,

V = volume in ml of ethanolic potassium hydroxide solution used in the determination,

N = the normality of the ethanolic potassium hydroxide solution used, and

m = mass in g of the sample.

Express results to the first decimal place.

3.2 Free Hydroxylamine Method — Method B

3.2.0 *Outline of the Method* — The carbonyl compounds are converted into oxime by reaction with hydroxylamine freed through the action of a solution of potassium hydroxide on the hydroxylammonium chloride. The hydroxylamine remaining after the reaction is determined by titration with standard hydrochloric acid.

NOTE — This method is suitable for perfumery material containing aldehydes including citronellal and certain ketones, in the absence of certain amount of ester of other alkali sensitive constituents. The method to be used shall be stated in the specification for the oil.

3.2.1 *Apparatus*

3.2.1.1 *Alkali-resistant glass flask* — of capacity 100 to 200 ml, to which can be fitted by means of a ground-glass joint, a glass tube at least 1 m long and about 1 cm internal diameter, serving as a reflux condenser, where required or a glass stopper.

3.2.1.2 *Pipette* — 20-ml capacity.

3.2.1.3 *Conical flask* — 200-ml capacity, with ground-glass stopper.

3.2.1.4 *Burette* — 10-ml capacity, graduated in 0·1 ml.

3.2.2 *Reagents*

3.2.2.1 *Hydrochloric acid* — 0·5 N.

3.2.2.2 *Hydroxylammonium chloride solution* — *see* **3.1.2.3**.

3.2.2.3 *Bromophenol blue ethanolic solution* — *see* **3.1.2.2**.

3.2.2.4 *Potassium hydroxide solution* — *see* **3.1.2.1**.

3.2.3 *Procedure*

3.2.3.1 Weigh into a flask to the nearest 1 mg, a mass *m* of the perfumery material as prescribed in individual standard. Introduce by means of a pipette, 20 ml of hydroxylammonium chloride into a conical flask. Add 10 ml of potassium hydroxide solution measured from a burette. Mix it well.

Pour the liquid into the flask containing the perfumery material and reserve the conical flask without washing. In case of aldehydes, allow to stand for 15 minutes at room temperature while for ketones, reflux gently for one hour using a water condenser or an air condenser at least 75 cm long. Cool to room temperature (*see* Note below). Where the individual standard specifies, add bromophenol blue indicator (as for dark coloured perfumery materials).

NOTE — Some aldehydes or ketones have been found to require longer standing or heating. In such case the change will be noted under the individual standard.

Neutralize with hydrochloric acid contained in the burette till a greenish-yellow colour is observed. Transfer the liquid to the conical flask used to mix the reagent and the alcoholic potassium hydroxide solution, and pour about half back again into the flask. Continue the neutralization in one of the flask only till the greenish-yellow colour of the indicator changes to lemon-yellow. The colour obtained in both flasks, shall not alter further when two drops of 0·5 N hydrochloric acid are added.

Alternatively this titration may be carried out by a potentiometric method to *p*H **3.5**.

3.2.3.2 *Blank determination* — Simultaneously with the determination prescribed in **3.2.3**, carry out a blank test with the same reagents and following the same procedure but omitting the perfumery material.

3.2.4 *Calculation*

3.2.4.1 The carbonyl value is calculated as follows:

$$\text{Carbonyl value} = \frac{56{\cdot}1 \times (V_1 - V_2) N}{m}$$

where

V_1 = volume in ml of standard hydrochloric acid used in the blank titration,

V_2 = volume in ml of standard hydrochloric acid used in the determination,

N = the normality of the hydrochloric acid used for titration, and

m = mass in g of the sample.

3.2.4.2 The content of carbonyl compounds expressed in terms of designated aldehydes and ketones is calculated as follows:

$$\begin{array}{l}\text{Content of carbonyl}\\ \quad\text{compounds (as aldehydes}\\ \quad\text{and ketones), percent}\\ \quad\text{by mass}\end{array} = \frac{M (V_1 - V_2)}{20\, m}$$

where

M = molecular mass of the aldehyde or ketones specified in the individual standard for the particular perfumery material,

V_1 = volume in ml of standard hydrochloric acid used in the blank titration,

V_2 = volume in ml of standard hydrochloric acid used in the determination, and

m = mass in g of the sample taken.

Express the results to the first place of decimal.

NOTE — In this, it is assumed that the alohols are monohydric and acids monobasic.

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